Synthesis and characterization of $YBa_2Cu_3O_{7-x}$ superconductor

Thesis submitted in the partial fulfilment of the requirements for the award of the degree of

MASTER OF SCIENCE

By

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CERTIFICATE

This is to certify that the work in the thesis entitled “**Synthesis and characterization of YBCO superconductor**” submitted by Chandan Mahto, in partial fulfilment of the degree of Master of Science in Physics at National Institute of Technology, Rourkela, is an authentic work carried out by him under my supervision and guidance. The work is satisfactory to the best of my knowledge.

DATE:

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ABSTRACT

We have synthesized YBCO superconductor via solid state reaction route and characterized it with XRD, R vs. T and I-V measurements. From XRD graph we have identifying the phase formation of our sample and from R vs. T measurement we found the onset of superconductive transition occur at (T_{CO}) 92.17 K; which also gives an extra supporting data towards the phase formation. We also subjected our sample for I-V characterization at different temperatures (30, 35, 45, 50 K) through four probe method and able to find out the critical current density at the subsequent temperatures and found a decrement of critical current density with increment of temperature.

CONTENTS

CHAPTER -1

1. INTRODUCTION

1.1 Superconductors

The Dutch physicist H. K. Onnes found that electrical conductivity of some metal and alloys approaches infinite value at low temperatures that means the resistivity is almost zero at that temperature. This phenomenon is called superconductivity and the material is known as superconductor. Many metals like Pb, Al, Sn, Nb, etc shows zero resistivity at around 4K.

The temperature at which a material shows zero resistivity is known as the transition temperature (T_C).The superconductors undergoes a phase transition from normal resistivity to zero resistivity at T_C. That is why T_C is called superconducting transition temperature. Many metals, alloys and compounds exhibits superconductivity but good conductors like Ag, Au, and Cu are not superconductors.

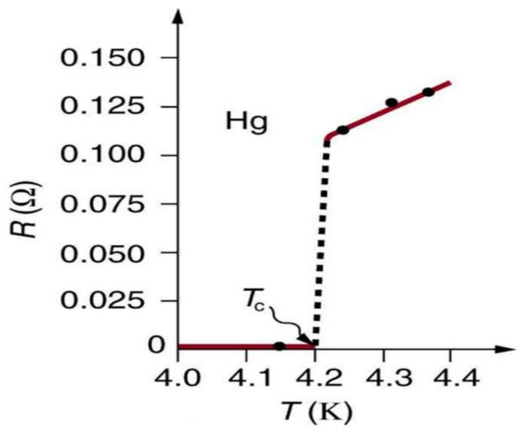


Fig 1- Resistivity – Temperature variation of Hg

1.2 Properties of superconductors

1. Materials having high normal resistivity exhibit superconductivity.
2. Superconductivity is exhibited by metallic elements in which the number of valence electrons lies between 2 and 8.
3. Transition temperature T_C varies from sample to sample.
4. The transition range for chemically pure and structurally perfect specimen is very small but impure and structurally imperfect is broad.

5. Transition metals having odd number of valence electrons are better than those having even number of valence electrons as regards superconductivity is concerned.
6. Elements with small atomic volume and atomic mass are better superconductors.
7. T_c varies linearly with Z^2, where Z is the number of valence electrons.
8. Ferromagnetic and antiferromagnetic materials do not show superconductivity.
9. In a superconductivity ring, current persists for a long time.

1.3 Effect of magnetic field

If the temperature T of the specimen is raised above T_C the superconductivity disappears. It also disappears if the magnetic field H is raised above the critical magnetic field H_C, where h is the function of T.

If $T = T_C$, then $H = 0$

If $T < T_C$, H_C increases. The variation of H_C with temperature is given by

$$H_C(T) = H_C(0)[\, 1 - (T/T_C)^2 \,]$$

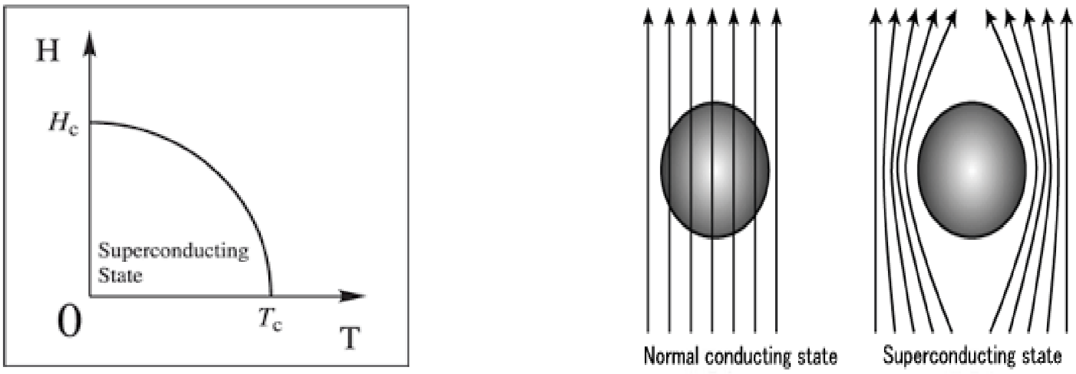


Fig 2 – Magnetic field Vs Temperature and Meissner effect.

1.4 Type I and Type II superconductors

On the basis of diamagnetic response of material, superconductors are of two types, type I and type II. Type I superconductors are those for which Meissner effect is complete, i.e., perfect diamagnetism. Below critical magnetic field(H_C),if the magnetic field H gradually increased from its initial value the magnetization M increases at $H = H_C$, the diamagnetism abruptly disappear. In type II superconductor when the magnetic field is increased from $H = 0$ to $H = H_{C1}$, the material behaves as pure superconductor and line of flux rejected. If h increased beyond H_{C1} the material is in mixed state up to H_{C2} and flux begins penetrating, Meissner effect is incomplete in this region.

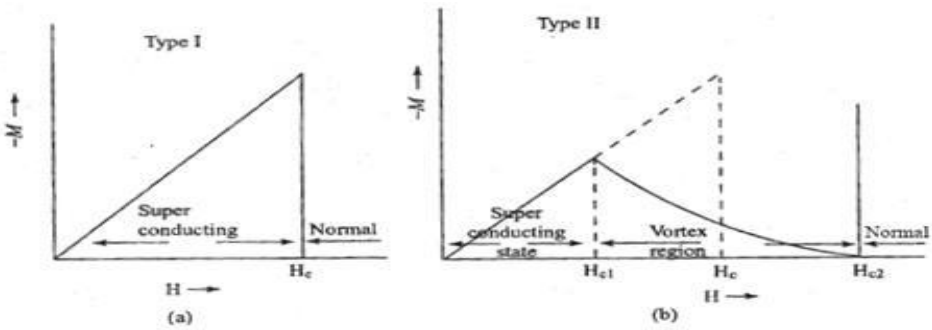


Fig 3 – Type I and Type II superconductor

1.5 BCS (Bardeen-Cooper-Schrieffer) theory of superconductor

To designate the superconducting occurrences a microscopic theory was suggested by Bardeen, Cooper, and Schrieffer known as BCS theory. In superconducting state the electrons form bound pair called Cooper. The Cooper pair basically formed due to the electron-electron interaction with an exchange of phonon. If we consider that the conduction electrons inside the Fermi sphere and the two electrons lying just inside the Fermi surface and they form pair of electrons. The Fermi surface electrons travels faster in the lattice. The positive core is attracted from the electrons. Due to the screening effect of the positive charges, the electronic charges is decreases. In this region electrons are in stable position takes enough positive charges in normal state that the lattice becomes distorted. During the formation of Cooper pair first electron release a phonon which is absorbed by the other electron. When the temperature is less than the transition temperature, the lattice-electron interaction is greater than electron-electron in that temperature range. The Cooper pair travels easily in the lattice with constant momentum and without any exchange of energy to the lattice meanwhile the pair gathering contains high degree of association in their motion.

1.6 Applications of superconductivity

1. *Switching devices*:- The transition from superconducting state to normal state when the applied magnetic field be larger than the transition field and is opposite when the field is removed. We have use this property to make switching element cryotron.
2. *Electric generators*:- The superconductor coil is winding in an top of greater magnetic field to create electric powers. This generators at very low voltage (450

V) could be generate very high powers (nearly 2500 kV). From that generator we can save the power and the size and weight of the generator is very low.

3. *Generation of magnetic field*:- The application of superconducting materials that can be generates high magnetic field (nearly 50 T). The power require to such a high magnetic field is only 10 kW but in general to generate such a high magnetic field require 3 MW power.
4. *Low loss transmission line and transformers*:- The resistance of the superconductor nearly zero so in superconducting wire power loss is very small. In transformer if the primary and secondary winding by a superconducting wire so that heat will be minimum and power loss will be very less.
5. *Magnetic levitation*:- The superconducting materials exhibits diamagnetic behaviour that rejects all the magnetic field lines and this is used in magnetic levitation. The magnetic levitation can be utilised in high speed transportation.
6. *Magnetic resonance imaging (MRI)*:- MRI is depend on the property of superconductivity and in a magnetic field there is a special type of behaviour of atoms. In human body there are large number of hydrogen atoms and other elements. The nuclei of atoms tend to aligned like a compass needle in the attendance of magnetic field.

CHAPTER 2

LITERATURE SURVEY

2.1 High temperature superconductors

Superconductors have huge number of conceivable useful applications but due to the limitation of low critical temperature applications are limited. Due to low critical temperature the repairs of conductivity through cooling requires very high cost. So that the researchers tried to find out the high critical temperature superconductors.

Bednorz and Muller in 1986 found a new ceramic oxide superconductor with transition temperature 30 K. These are known as high-T_C superconductors (HTSC). The first HTCS was $La_{2-x}M_xCuO$ (x = Ba, Sr, Ca) with transition temperature between 20 K to 40 K. They are called 214 system with K_2NiF_4 structure and orthorhombic misrepresentation.The another HTCS is $LnBa_2Cu_3O_{7-x}$ (Ln = Y, Nd, Sm, Yb, Gd, Ho, Dy, Tm, Er) and is known as 123 system with orthorhombic structure.

The origin of the mechanism of high temperature superconductor is until now investigate. In high temperature superconductors oxygen is play important role. The real conduction tool of high temperature is feasibly most challenging task in condensed matter physics recent days.

2.2 Properties of High Temperature superconductors

Structural properties

The high temperature superconductors have ideal perovskite structure whichever from side to side an intergrowth phenomenon or by a systematic elimination of oxygen atoms. High temperature conductors have layered crystal structure more than one CuO_2 layers, the copper atom is strongly and covalent bonded close to the arrangement of square planar of four oxygen atoms. In high temperature the conduction of the charge and from CuO_2 layers takes place because of doping close to the metal insulator phase.

Physical properties

1. In general oxide high temperature superconductor the conduction charge carrier is holes rather than electron.

2. The magnetism and superconductivity are hardly found in earth compound and in the process of superconductivity the earth ions does not contribute.
3. In superconducting state of low temperature the specific heat is

$$C = aT^{-2} + bT^{-3} + cT$$

   The first term is due to the Schottky like irregularity, the second term due to lattice of Debye contribution and the third term because of conduction of free electron.
4. All the high temperature superconductor are type II superconductor, highly anisotropic with H_{c1} and H_{c2}.
5. When the pressure increases in high temperature superconductors the critical temperature will be increases, however reverse effect is found in predictable superconductors.
6. In high temperature superconductors does not present the isotopic effect.
7. Temperature dependent and highly anisotropic conduction properties in normal state.
8. The Josephson tunnelling in high temperature superconductors

2.3 Application of high temperature oxide superconductors

1. The high temperature superconductors are used in very large scale integration technology and it is predictable that in future used in high speed computer and telecommunication.
2. In this days used in sensitive magnetometers from SQUIDs working. But SQUIDs made of oxide superconductors working at 77 K and are predictable to progress the roughness and usefulness of the device. This will also major application in soldierly and medicine.
3. The high temperature superconductors have significant used in transmission, generation, storage, transformation of electrical power to electrical industry.

2.4 YBCO superconductor

YBCO is a high temperature superconductor whose transition temperature start at 93 K and zero resistance below the onset temperature of Tc. YBCO is a type II superconductor in that it has both Meissner effect and intermediate state. However, there are certain changes in the way the intermediate state operates.

In YBCO the cooper pairs as being the pairing of holes rather than electrons. Using holes as a charge carrier means that the charges that move are positive rather than negative. Holes in the cuprate superconductors come from the C_u^{2+} and C_u^{3+} states that are present. The number of holes copper oxide conduction planes is prejudiced by the ratio of these two states. This ratio can be altered by the quantity of oxygen present in the planes. The superconductive state is be contingent on the awareness is met. This is why changing the oxygen gratified in YBCO differs the critical temperature since this is done the concentration of holes is affected.

2.5 Structure of YBCO

The structure of YBCO acts a significant role in superconductivity. YBCO has a layered structure containing copper oxygen planes with Yttrium and Barium atoms in the crystal structure in addition. The subsequent crystal structure is related to a pervoskite with a unit cell involving of fixed cube of $BaCuO_3$ and $YCuO_3$. In figure shows the structure and the chemical arrangement of the planes formed. One significant entity to note that is the two unlike planes of copper and oxygen. The planes below and above the Yttrium atoms have two oxygen atom per copper where Yttrium has planes close it with one copper per oxygen. These planes that are one to one are said to be oxygen lacking since when related to a whole perovskite structure there are two oxygen atoms omitted.

The superconducting of the system appears to rise from these copper oxide layer since they are mutual to the copper oxide superconductors. The two planes detached by an atoms of Yttrium, a distance of 3.2 A. The current runs through the two CuO_2 planes. The distance between the copper atom in the in these planes make it easier for charge to expectation between ions than from plane to plane. In between these conduction layers there are Barium, Yttrium and supplementary copper oxygen pairs. However these layers are not where current runs through the material, they play an important part in superconductivity.

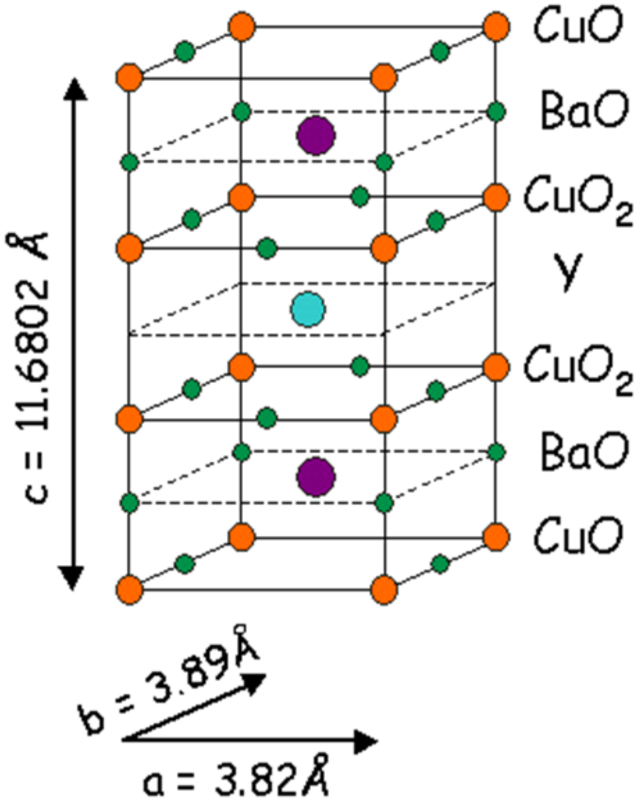


Fig. 4. Structure of YBCO

2.6 Defects of YBCO

YBCO is a high temperature superconductor and till the discovered the high temperature superconductor structure is very complex and sophisticated feature comparing with all the other superconductors. There are many atoms bonded such a manner that changes the properties of the superconductors, especially in copper oxide superconductors. After the discovery of cuprate superconductors explains that the defects plays significant role for governing the superconducting parameter of the system. In YBCO the defect is due to the oxygen deficiency in the crystal structure. The basal plane structure of the chain of CuO - CuO and between the Cu ions there are no any oxygen. Most of the materials defects can be increased by the doping but in the high temperature superconductor result is surprising that the defects are varying due the structure of CuO_2 conduction planes. The structural transition from orthorhombic to tetragonal is also happen due to the oxygen deficiency in the high temperature superconductors.

2.7 Vortex pinning effect

There is a small gap between the normal and superconducting state is called the vortex state or mixed state. The normal, vortex and superconducting are basically in the type II superconductors. The normal core set in superconducting materials. In the normal state of microscopic is encircled by the conducting currents. In the normal states, the field penetrates of the of vortex lines in the mixed or vortex state. The every vortex lines has a core radius. The field is maximum at the centre of materials and it is falls to 1/e times of the maximum field at the distance from the centre. The flux vertex is known as fluxoid. The fluxoid brings quantum magnetic flux.

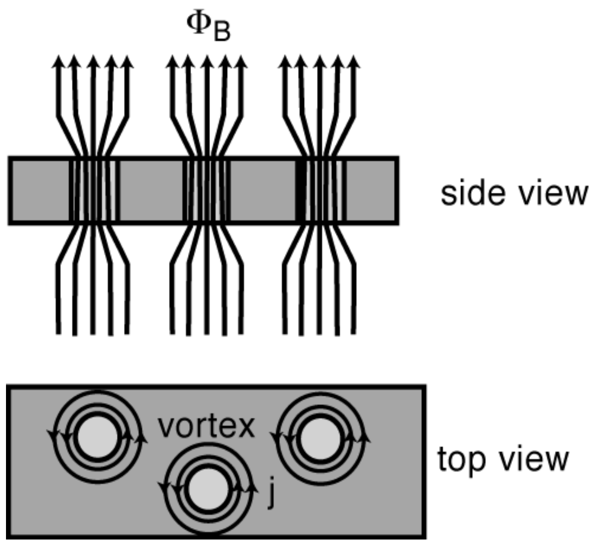


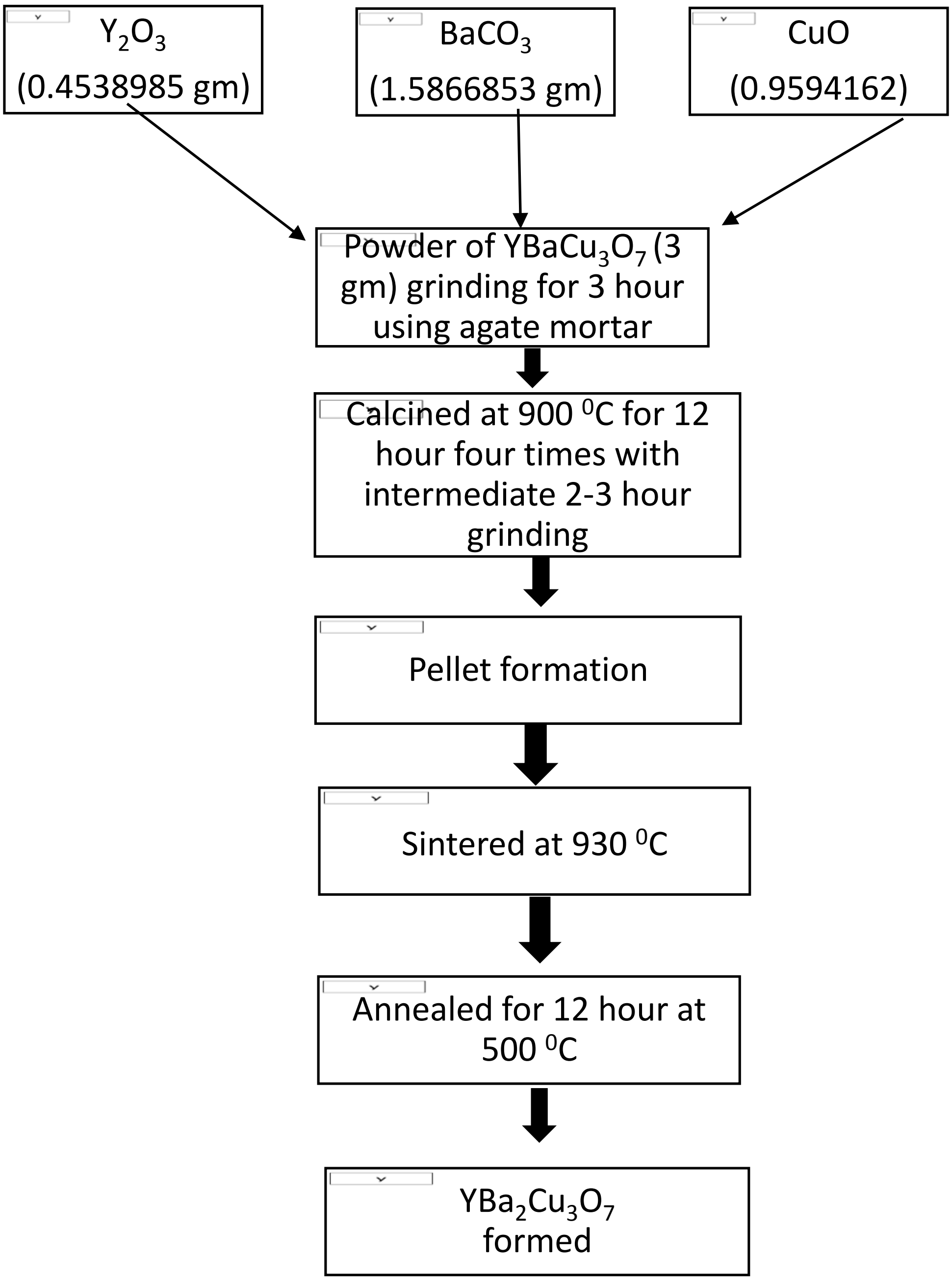
Fig.5. pinning effect

The idea of the vortex in ideal materials would be equal spacing. The crystal inhomogeneous in the real materials that's why the formation of vortex in the superconductivity. The question arises that at the mixed state the still take into account the perfect conductivity. The Lorentz force is acting on the vortex or pinning sites in mixed state due to the inhomogeneous of the real materials. The dissipation force is due the vortex motion governed to destruction of superconductivity. The understanding of the role of the fluctuation in the superconductor may be destroy the superconducting state. The fluctuation effect is a static property which depend on the electrical and thermal conduction of the planes. According to the Ginzberg and Landau theory the electron density of the superconductor is proportional to the magnitude square of the complex wave function in the superconducting state. From this theory the flux ϕ_0 = hc/2e, 2e is due to the conduction of the Cooper pair. If the magnetic field is applied the superconducting rings are in normal state. When cooling the ring lower than the transition temperature, the flux is excluded from the ring but passes through the hole. If the external applied magnetic field off the flux through the hole remains trapped. The super- current generated around the ring maintain the flux through the hole of the ring.

CHAPTER -3

EXPERIMENTAL PROCEDURE

3.1 Synthesis of YBCO superconductor:



Stochiometric amount of Yttrium Oxide, Barium Carbonate and Copper Oxide was taken as precursers to obtain the desire material YBCO.

$$Y_2O_3 + 2BaCO_3 + 3CuO \longrightarrow 2YBa_2Cu_3O_7$$

The ingredients were grounded together in an agate mortar for 2-3 hrs to obtain a homogeneous mixture.

After grinding, the powder was calcined at 900 ^{0}C in a muffle furnace for 12 hour and then the calcined powder was again heated for 4-5 times with intermediate grinding at the same temp.. After repeated heating, the resultant powder obtained, is pressed into pellets of 1 mm thickness and finally sintered at 930 ^{0}C for 12 hrs and followed by oxygen annealing for 12 hrs for Oxygen uptake and thus obtained the resultant YBCO.

CHAPTER-4

CHARACTERIZATION TECHNIQUES

4.1 X-ray diffraction technique

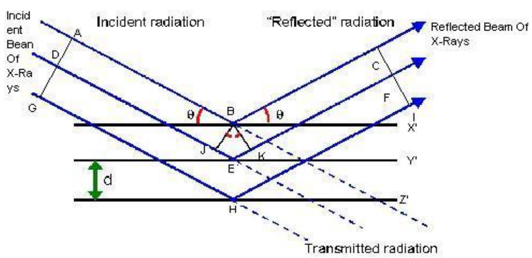
The X-rays are diffracted from the three dimensional space grating. The structure of the crystal successfully determine by X-ray diffraction technique. When the atom of the single crystal are diffracted when a momochromatic beam of X-ray incindent parallely. The direction of the beam and intensity is find out the lattice structure and chemical composition of the crystal. X-rays are electromagnetic waves with extremely small wavelengths (usually order of 10^{-10} m). X-ray diffraction technique is used to analyze the crystal structure because the wavelength of X-ray radiation is very close to the interplanar spacing of most of the materials. The XRD technique is based on the principles of constructive interference.

Bragg's law describes the relation between the angle at which x-ray of particular wavelength diffracts and the wave length. The phase composition of a material can be determined by the help of XRD

$$2d\sin\theta = n\lambda$$

where d- inter planar spacing and θ- Scattering angle and n- Represents the order of diffraction ; λ – Wavelength of x radiation

When the monochromatic X-rays falls on the plane of the crystal of atoms such that the angle of reflection is equal to the angle of incidence. Due to the set of parallel planes the constructive inerference beams are reflected and provide to a strong diffraction beam. The



wavelength remains same due to the elastic scattering of atoms with the X-rays.

Fig.6. Schematic diagram of Bragg's diffraction

4.2 Resistivity-Temperature measurement technique

Resistance can be measured by various methods, one of the methods being the four probe technique. If there is any temperature gradient in the system, then voltage will arise in different parts of the circuit and also the sample. This phenomenon should be compensated. In order to do this, the voltage should be measured with applied current and differences taken and also without them. Dc four probe techniques for this is the best method. In two probe technique, there is a chance of error due to contact resistance. But, in case of four probe technique, both contact resistance and lead resistance are eliminated.

Now, while making electric connections to the sample, it should be taken care of that the contact resistance should be kept as low as possible. Contact resistance is basically the resistance between the electric wires and the sample. If the contact resistance is high, there would be more noise and errors in the result will be visible. The contacts should be given a suitable shape. Stability is a very important factor in this case. For the sake of experiment, several measurements are needed to be taken and it should be made sure that the contacts remain stable over time.

Resistance Temperature Detectors are temperature sensors that have a resistor that varies resistance value as its temperature increases or decreases. They have been applied in many purposes, for many years to measure temperature and have developed a name for precision, stability and repeatability. The material has a predictable change in resistance as the temperature changes; it is this predictable change that is used to determine temperature.

Resistance thermometers are made in a number of forms. They proffer greater stability and accuracy. Resistance thermometers use electrical resistance and need a power source to function. The resistance changes nearly with temperature. Lead wire resistance can also be a issue; adopting three- and four-wire, instead of two-wire, connections can eradicate connection lead resistance effects from measurements. Three-wire connection is adequate for most purposes. Four-wire connections are used for the most accurate applications.

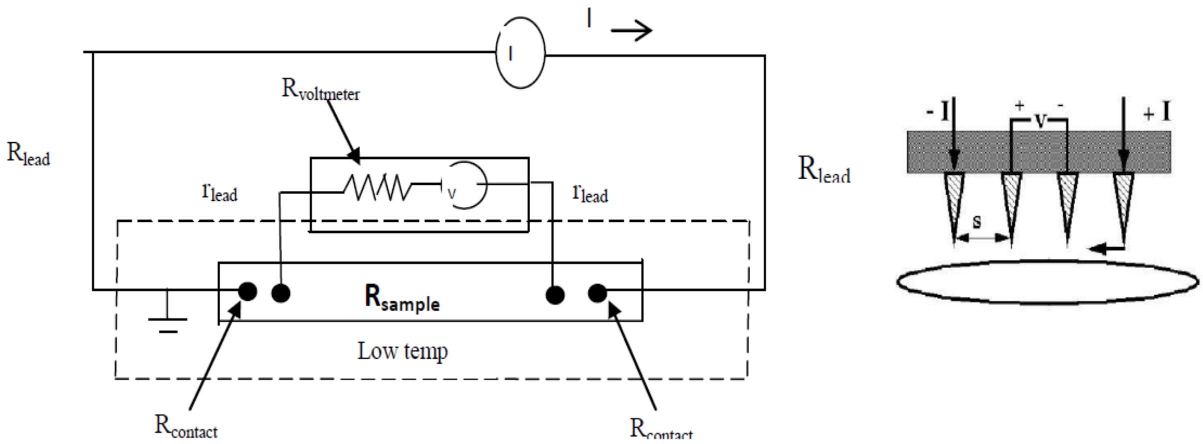


Fig.7. – Diagram of four probe

Resistivity be governed by the nature of the material. When a current of none value is nourished to the material, then the potential difference is generated in the voltage probe via point connection. From this measurement the resistivity is calculated.

We use four probe method because it decrease the other influence i.e., lead resistance, contact resistance etc to the resistance measurment which results an precise measurement of the material resistance. The external two probe are used for finding the current and two inner probes are used for calculating the ensuing voltage drop across the the surface of the material.

The voltage $V = (I - I_1)(R_{sample}) - I(2r_{contact} + 2r_{lead})$,As R_{volt} is much larger then other resistance in the cicuit. $I_1 << I$, so $V = IR_{sample}$

4.3 I-V measurement technique

The experimental set up for performing I-V measurement was same as in the case of R-T measurement; but the purpose of measurement of this quiet different. Here we measure current against the voltage at fixed temperature to calculate a most valuable parameter J_c, which is the critical current density. The intercept of the I-V curve will give me critical current I_0 and the by using the formula current per unit area we can calculate J_c at different temperature from the sample in the superconducting state.

CHAPTER -5

RESULT AND DISCUSSION

5.1 XRD analysis

From the XRD studies we found all peaks match with the standard data with a few no. of impurity peaks which are always present in the sample as it is a complex phase. So for surety of the phase we did the R-T measurement of the sample.

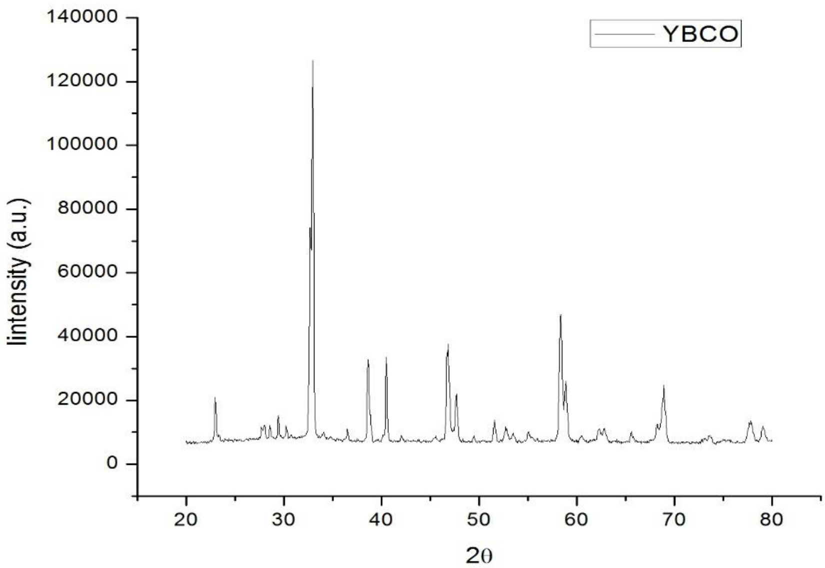


Fig.8- Graph plot between intensity and 2 theta

5.2 R-T anlysis

The temperature dependence of resistivity is measured using standard four probe technique by using closed cycle refrigerator. From the plot obtained between resistivity versus temperature from 300K down to around 40 K. We found the onset of superconductivity around(T_{CO}) 92.17 K and the critical temp. T_C at around 90 K reveals that there are certain impurities in our sample.

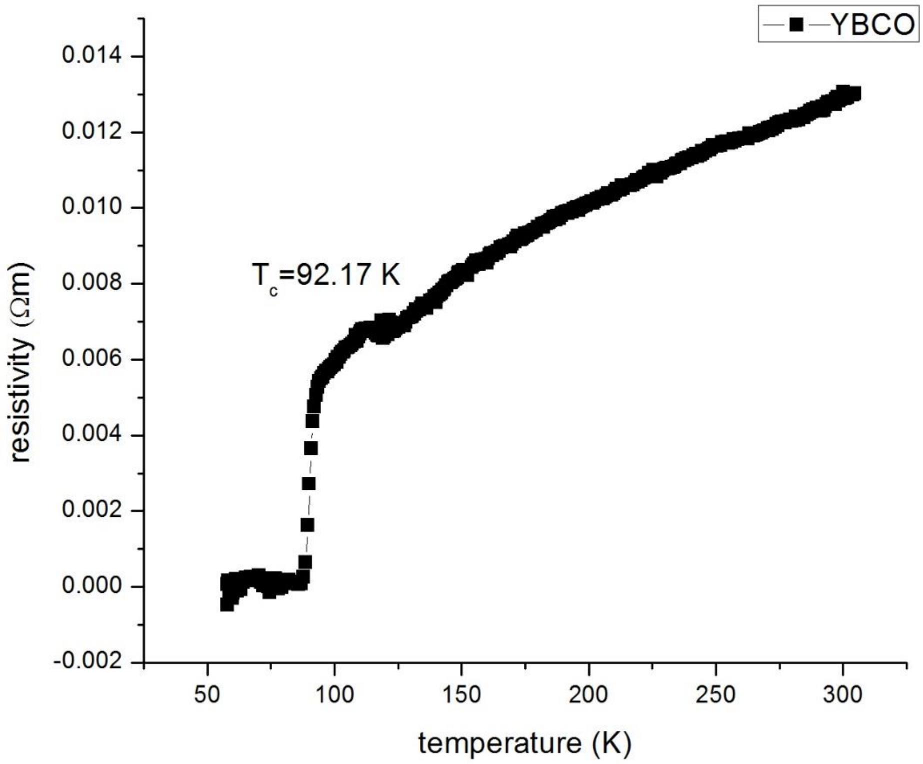


Fig.9 – Resistivity-Temperature of YBCO

5.3 I-V analysis

In this measurement we applied current across two probes and voltage was measured at constant temperatures with the help of close cycle referigerator. The current was varying with the current source and calculated their corresponds voltage.From the figures at different temperatures the critical current I_c is calculated. From the figures 10-14 the graph between current and voltage , we are able to calculate critical current corresponding to different temperatures (30K, 35K, 45K 50K).From the figure, we observed an increment of critical current density with decrement of temperature which is clearly shown in figure-15.

TEMPERATURE(K)	I_c= CRITICAL CURRENT(mA)	Jc=CURRENT DENSITY (A/m^2)
30	80	1.5814×10^3
35	59.95	1.185×10^3
45	52.27	1.0332×10^3
50	41.38	0.818×10^3

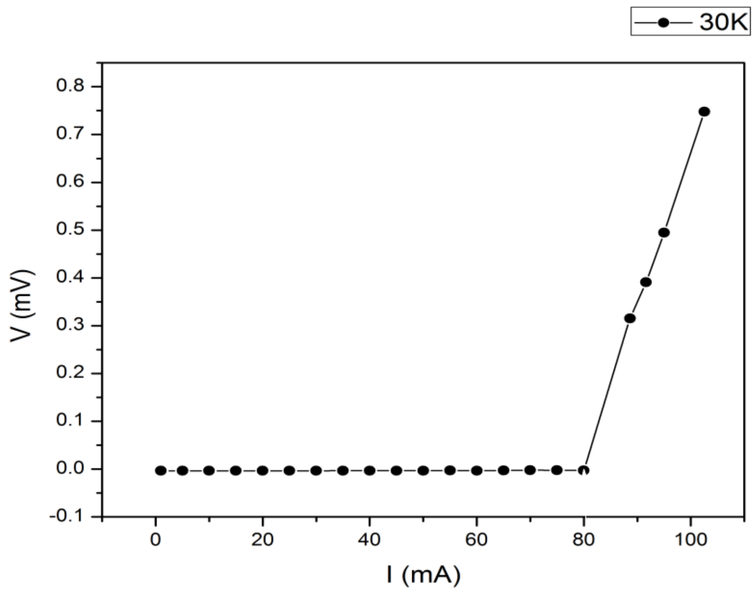


Fig.10 – Graph between Current vs Voltage at 30 K

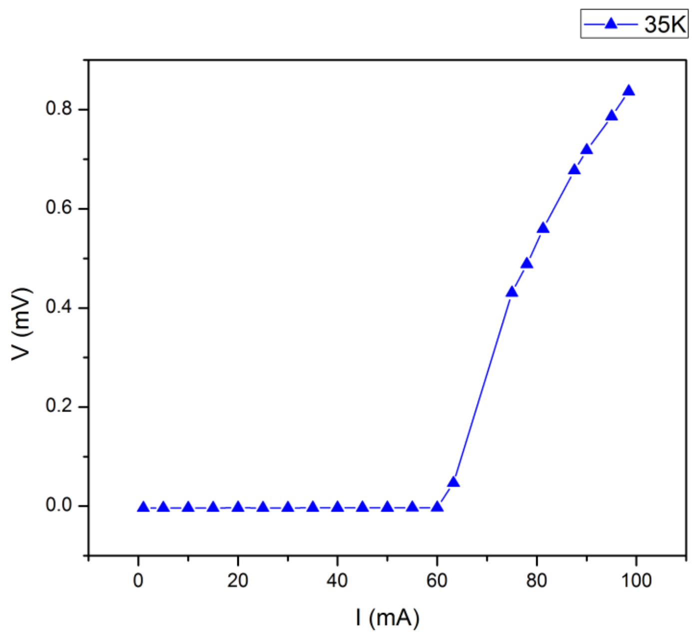


Fig.11 – Graph between Current vs Voltage at 35 K

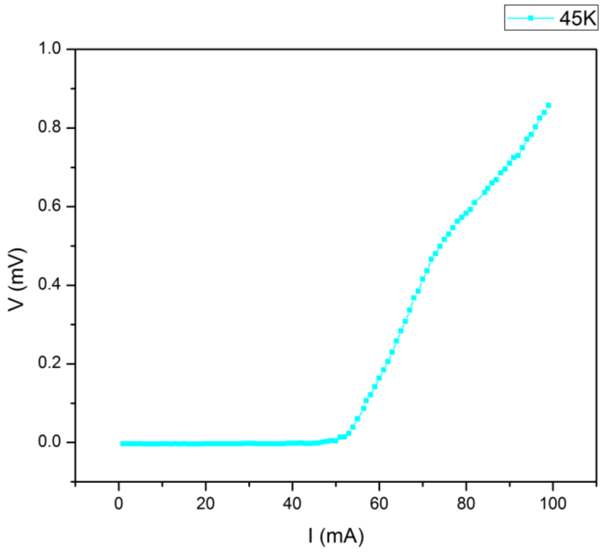


Fig.12 – Graph between Current vs Voltage at 45 K

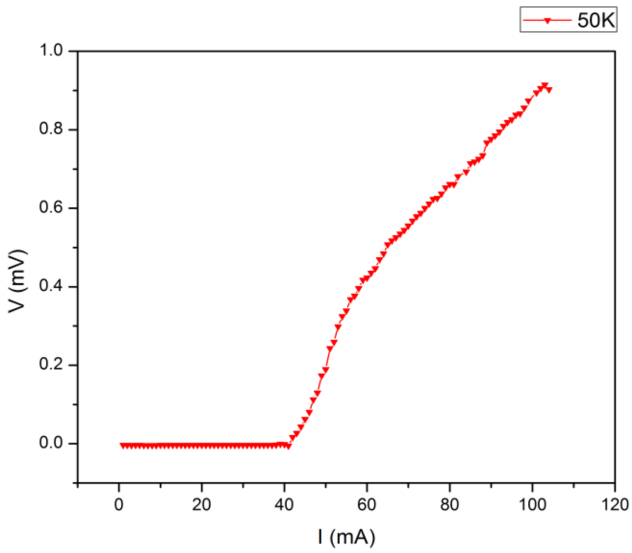


Fig.13 – Graph between Current vs Voltage at 50 K

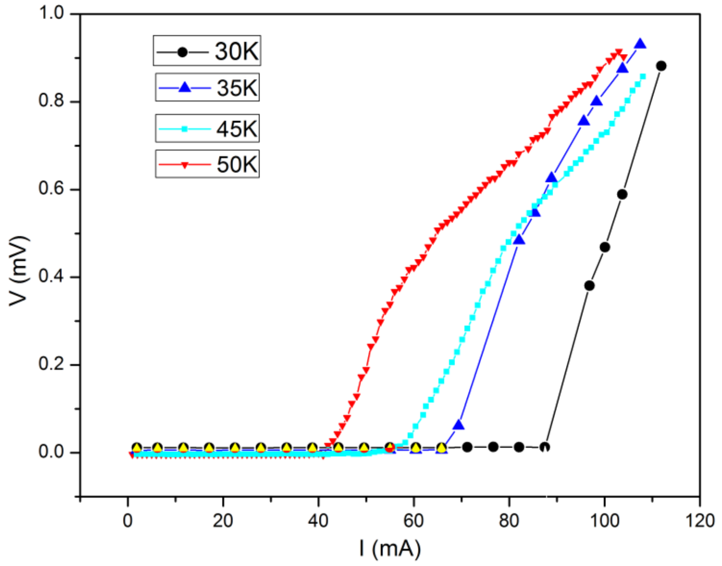


Fig.14 – Comparision of graph between Current vs Voltage for all temperatures

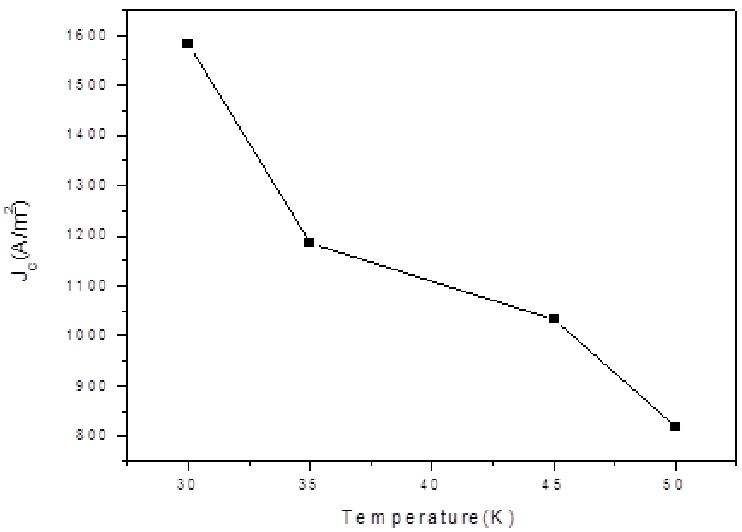


Fig.15- Graph between critical current density and temperature

CHAPTER-6

CONCLUSIONS

The YBCO superconductor is prepared successfully via solid state reaction method. From XRD graph we are confirmed the phase formation of YBCO sample. From the RT measurement we found that the transition from normal state to superconductive state around 90 K, which also gives an extra supporting data towards the phase formation .We also subjected our sample for IV characterization at different temperatures (30, 35, 45, 50K) via four probe method and able to find the critical current density at the subsequent temperatures and found a decrement of critical current density with increment of temperature.

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